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Effect of SLM Parameters on the Structure and Properties of CP-Ti

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Abstract. Selective laser melting (SLM) is an additive manufacturing technique, which allows varying the relative porosity at the micro and macro levels. In this study, the microstructure, including the porous structure, of cp-Ti Grade 1 ELI produced by SLM and the effect of the scanning parameters on mechanical properties are studied. The mechanical properties are analyzed in terms of ISO 5832-2 for metal implants for surgery. The cp-Ti samples processed by SLM are characterized by increased strength due to the low porosity condition and the acicular martensitic structure. The ductility data scattering is explained by the stratified distribution of porosity in the metal after SLM.

INTRODUCTION

Additive manufacturing methods for obtaining blanks and finished parts suggest a layer-by-layer deposition of the workpiece contour using powder as a raw material, while the process is controlled by software, which allows a desired product architecture to be obtained. Titanium and its alloys play an important role in the formation of the market of new products manufactured by additive technologies [1, 2]. In particular, the medical use of titanium in the production of multipurpose implants is an actively developing field [3, 4, 5]. 3D printing of a finished product generally involves sintering or melting of metal powder depending on the method applied. The processing of powders is associated with such concepts as porosity (or density). In turn, these parameters are responsible largely for the physical and mechanical properties of the resulting product [6].

Two types of pores can be distinguished in additive-manufactured products. The occurrence of the first type of pores usually takes place at the microscopic level and inevitably accompanies the process of obtaining products from a powder. Therefore, it depends on the technological parameters of the process. The second type – macropores can be artificially set for the purposive tailoring the physical-mechanical properties, for example, the modulus of elasticity or strength. Regularly repeating macropores form the cellular structure of the material [7, 8, 9]. The mechanical behavior of porous/cellular materials can be considerably different. Therefore, special test methods have been developed to estimate their properties [10].

The purpose of this study is to estimate the effect of 3D-printing parameters on the microstructure including pores and the properties of solid products made of commercially pure titanium (CP-Ti).

MATERIAL AND METHODS

The raw material used for this investigation was the powder of Grade 1 cp-Ti with extra-low interstitial (ELI) content. The powder produced by a plasma atomization process was spherical with a particle size ranging between 15 and 50 with the highest frequency of about 35 μm. The chemical composition of the powder was determined using an ARL 9900 X-ray fluorescence spectrometer (wt%.): 99.8Ti, 0.07 Ce, 0.04 Nd, 0.04 Fe, 0.03 Mg, 0.02 Pr,

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0.01 Cr, 0.002-0.005 of Mn, Ni, P, and Si. The oxygen content of 0.10 wt% was estimated on a Horiba EMGA-620W gas analyzer.

The samples for mechanical properties tests were produced using layer-by-layer synthesis on a MeltMaster3D-550 selective laser melting (SLM) machine manufactured by JSC RPA CNIITMASH. The optimization of the SLM regimes implied three batches due to the limited space of the SLM machine. The following parameters were set the same in all the three batches: the laser spot diameter was estimated to lie in the range of 60...70 µm, the powder layer thickness was set to be 50 µm, and the protective atmosphere was argon (oxygen content up to 900 ppm). The cylindrical billets with a diameter of 12 mm were produced by SLM with the building direction (BD) orthogonal to the longitudinal axis of the billet. The sample density was measured by hydrostatic water weighing in accordance with GOST 15139 on a Shimadzu AUW-120D balance. The following SLM parameters were varied: a laser radiation power of 220 to 340 W, a scan speed of 700 to 900 mm/s, and a hatching spacing of 100 to 140 µm.

The purpose of the investigation was to produce a material with different porosity at the microlevel and to study the effect of porosity on the mechanical properties. The tensile test samples were made from the cylinder billets. The tensile tests were performed on an Instron 3382 machine at a grip displacement speed of 5 mm/min. The structure of the SLM samples was studied using an Olympus GX-51 light optical microscope.

RESULTS AND DISCUSSIONS

The sample volume for three batches and the process characteristics are given in Table 1. The increase in the scanning speed leads to a decrease in the yield strength. These data are qualitatively consistent with the results of the compression tests of SLM-produced cellular titanium samples reported in [11]. The difference in the proof strength values for different SLM conditions is shown in Fig. 1.

TABLE 1. The effect of SLM parameters on the average value of proof strength

Index		**Batch number**		
		1	**2**	**3**
Sample volume [pieces]		18	12	11
Average relative density [%]		95	98.3	98.2
Laser power [W]		220-280	220-280	250-340
Scanning speed [mm/s]		700-900	700-900	700-800
Hatch spacing [µm]		100-140	100-120	100-120
Average proof strength [MPa] at a scanning speed of	700 mm/s	476	538	551
	800 mm/s	-	-	536
	900 mm/s	340	509	

As can be seen from the bar chat, the proof strength for the samples of the second batch was higher than that for the samples of the first batch. The average relative density of the samples from the first and second batches was measured (95% and 98.3% respectively). Thus, an increase in the proof strength can be attributed to an increase in the density of the SLM metal. A maximum the relative density of 99.3% was obtained.

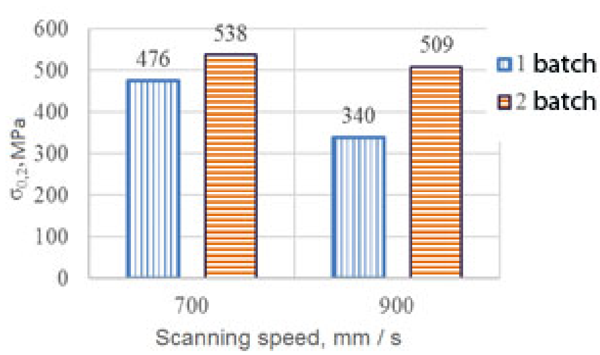


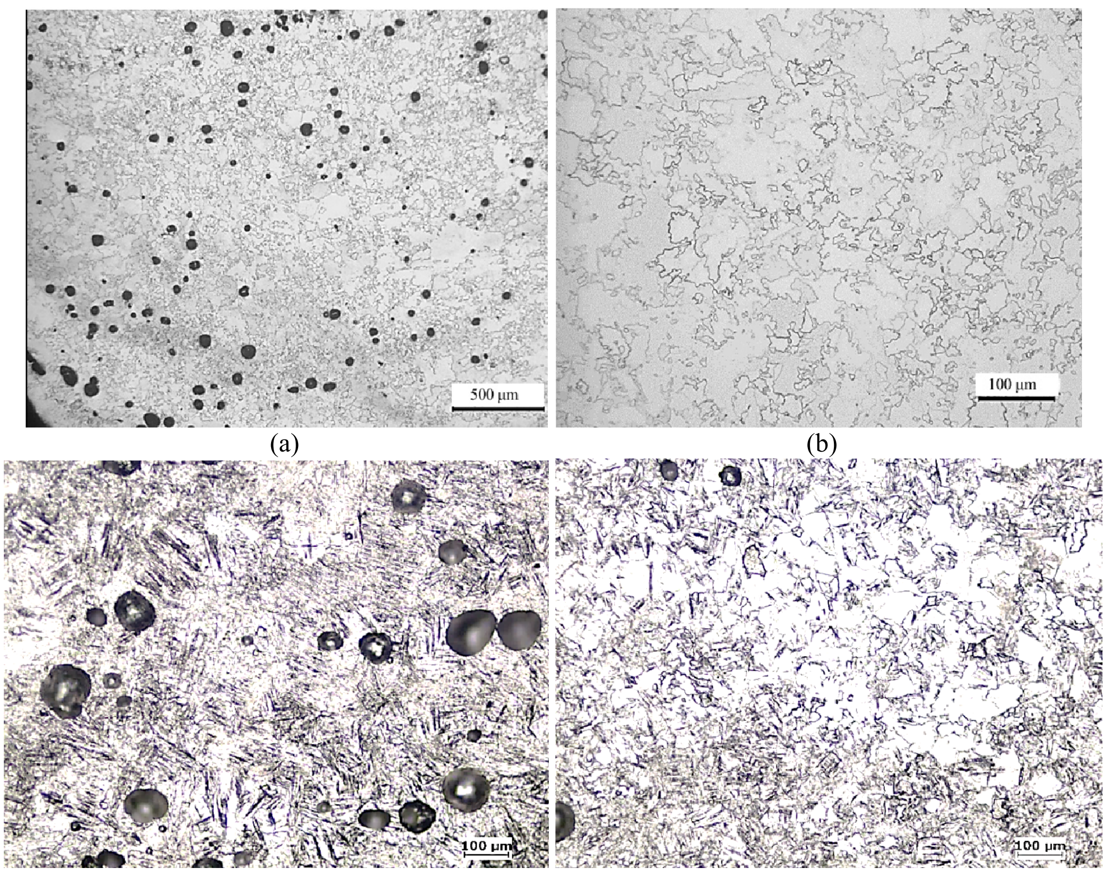
FIGURE 1. The effect of the scanning speed on the average values of proof strength

The most stable material properties were achieved in the third batch. The statistical data on mechanical properties are shown in Table 2.

TABLE 2. The effect of SLM parameters on the mechanical properties of the third batch samples

Parameter	Proof strength, MPa	Ultimate tensile strength, MPa	Elongation,%
Average	548	620	20
Standard error	7.0	5.4	0.9
Median	537	622	19
Standard deviation	23	18	3
Sample variance	542	328	9
Range	78	62	9
Minimum	512	584	17
Maximum	590	646	25

The oxygen content after SLM increased to 0.11 wt%, which passed cp-Ti from Grade 1 ELI to Grade 1 according to ISO 5832-2 "Implants for surgery. Metal materials". Table 2 demonstrates that a high average ultimate strength of 620 MPa was achieved, which is considerably higher than the strength values for annealed Grade 1 from ISO 5832-2, where the minimum allowable values of ultimate strength, proof strength and elongation are 240 MPa, 170 MPa and 24% respectively. However, the ductility was lower than the standard requirements.



(a) (b) (c) (d)

FIGURE 2. Microstructure of SLM-manufactured cp-Ti: a, b – the 1st batch; c, d – the 3rd batch

The gradation of ISO 5832 is quite inconsistent in terms of mechanical properties, since stronger materials are classified there as lower grades. For structural materials, the reverse approach is traditionally implemented. Thus, the ductility characteristics come to the fore in ISO 5832. According to it, for the premium grade ELI, the set of the above properties is 200 MPa, 140 MPa and 30%, respectively. Therefore, high strength is not a limiting parameter, especially since the materials are supplied in the annealed condition. The grade of the material in this case indicates a higher purity of titanium. The proximity of the average ultimate strength value of 620 MPa and the median value of 622 MPa observed in the experiments shows a symmetrical frequency curve and indicates that the strength distribution law is close to normal.

The data on the ductile properties of the samples (elongation before fracture, reduction area) is characterized by a considerable scattering of values. The authors of [12] indicated the possibility of such a result. They determined the mechanical properties of the samples as a function of their spatial location in a SLM chamber and of the sample building direction. Thus, the spread of the elongation values was up to 6%. As can be seen from Table 2, the spread of the elongation values in the present study is 9%, which is close to the literature data.

The strength properties vary between 62 and 78 MPa (Table 2), which is close to 100 MPa found in the literature data. The increased range of the mechanical properties is generally attributed to the defects unevenly distributed over the volume of the metal. These defects include micropores as well. Since the relative density of the material produced by SLM is not 100%, the presence of pores is obvious. The fluctuations in the mechanical characteristics may occur if the distribution of the pores is uneven or if their volume and/or shape are unequal.

Figure 2 demonstrates the microstructure of the samples from the first and third batches. The porosity of the samples with the same microstructure is stratified. Particularly, for the samples from the first batch, with the microstructure of α-phase grains close to equiaxed shape, regions with a sufficiently high porosity are observed along with regions with no visible pores. Likewise, layered porosity occurs in the samples from the third batch, which are characterized by an acicular martensitic microstructure formed under conditions of higher cooling rates.

CONCLUSIONS

Cylindrical billets with a relative density of more than 99% were produced from Grade 1 ELI cp-Ti powder by the SLM method. Despite the residual micro porosity, the ultimate tensile strength of the obtained material reached a considerably high value of 646 MPa. The increased proof strength was provided by the low porosity condition resulting from the reduced scanning speed and the acicular martensitic structure formed due to high cooling rate. The ductility was characterized by fluctuations, which is most likely due to the stratified distribution of the porosity of the metal with a layered structure.

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